



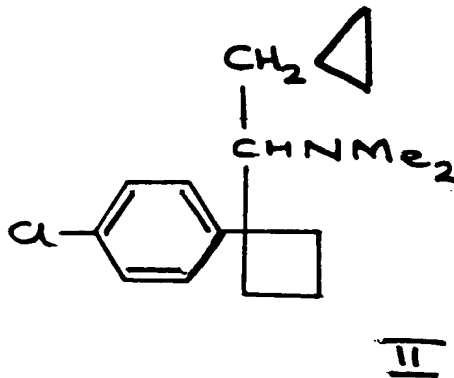
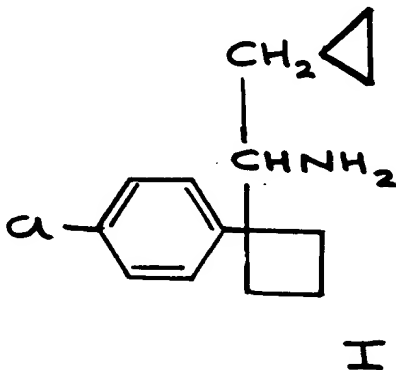
In the matter of US
Patent Application
Serial No. 725206
filed April 19th, 1985

#16

Declaration

I, Gerrard Haran, a British subject of 57 Zulla Road, Mapperley Park, Nottingham, England do hereby declare that:-

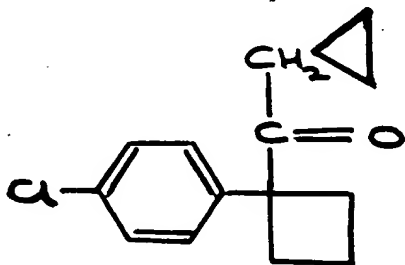
- 1) In 1969, I attained a B.Sc Hons. (Chemistry), from Glasgow University (Scotland) and in 1972, I completed a period of research in inorganic chemistry at Glasgow University (Scotland) which led to the award of a degree of Doctor of Philosophy.
- 2) I joined The Boots Company as a Research Chemist responsible for infra-red and nuclear magnetic resonance spectroscopy in 1974. In 1980, I was promoted to the post of Section Head of Physical Chemistry with the additional responsibility of gas chromatography. In this position I advise my colleagues within The Boots Company on the interpretation of nuclear magnetic resonance spectra.
- 3) In October 1984, at the request of Dr. J.E. Jeffery, staff under my direct control, using standard procedures, obtained proton nuclear magnetic resonance spectra of two compounds which were described in the above identified patent application as Examples 10(u) and 11(g) respectively. Structures I and II below had been assigned to these compounds at the time of their preparation in 1981. The compounds were in the form of hydrochloride salts.



The spectra obtained are attached hereto as Figures I and II.

4) I have examined these spectra and consider that signals at 6.00 δ to 5.48 δ which represent one proton and at 5.24 δ to 4.92 δ which represent two protons in the spectrum reproduced in Figure I and the signals at 6.10 δ to 5.54 δ which represent one proton and at 5.32 δ to 5.00 δ which represent two protons in the spectrum reproduced in Figure II show the presence in these two compounds of a $-\text{CH}=\text{CH}_2$ group. There are no signals at 0-1 δ which would indicate the presence of a cyclopropyl ring as shown in structures I and II. I therefore concluded that Structures I and II assigned to these compounds were incorrect.

5) In February 1986, at the request of Dr. J.E. Jeffery, staff under my direct control, using standard procedures, obtained a proton nuclear magnetic resonance spectrum (attached hereto as Figure III) of a ketone to which Structure III had been assigned at the time of its preparation in 1981.



III

The signals at 5.96 δ to 5.40 δ representing one proton and at 5.10 δ to 4.72 δ representing two protons show the presence of a $-\text{CH}=\text{CH}_2$ group. There are no signals at 0-1 δ which would indicate the presence of a cyclopropyl ring as shown in structure III. I therefore concluded that Structure III assigned to this ketone was incorrect.

Further declarant sayeth not

I, the undersigned declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States code and that such wilful false statements may jeopardise the validity of the application or any patent issuing thereon.

Gerrard Haran

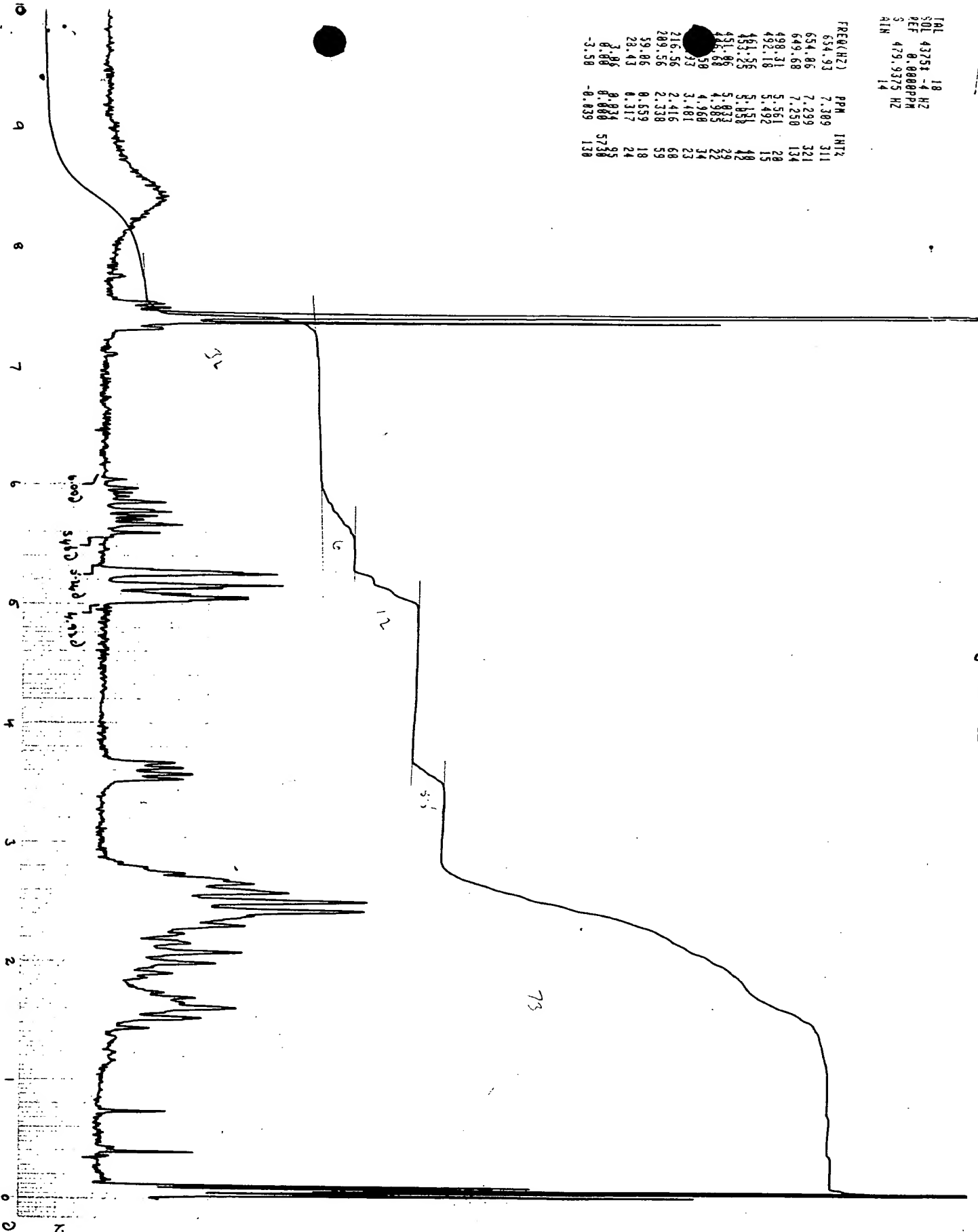
Gerrard Haran

Signed at Nottingham, England on the 24th day of March, 1987.

Figure I

18
 50L 43751 -4 HZ
 REF 0.000PPM
 5 479.9375 HZ
 14

FREQ(HZ)	PPM	INT.
654.93	7.309	311
654.86	7.299	321
649.68	7.250	134
498.31	5.561	28
492.16	5.492	15
481.25	5.451	42
471.86	5.405	29
466.68	4.985	34
450.50	4.700	23
216.56	3.481	68
209.56	2.338	59
59.86	0.559	18
23.43	0.317	24
8.88	0.024	573
8.88	0.000	5738
-3.58	-0.039	130



KC 82243

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C0043
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89.55
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2V
 19.10.84

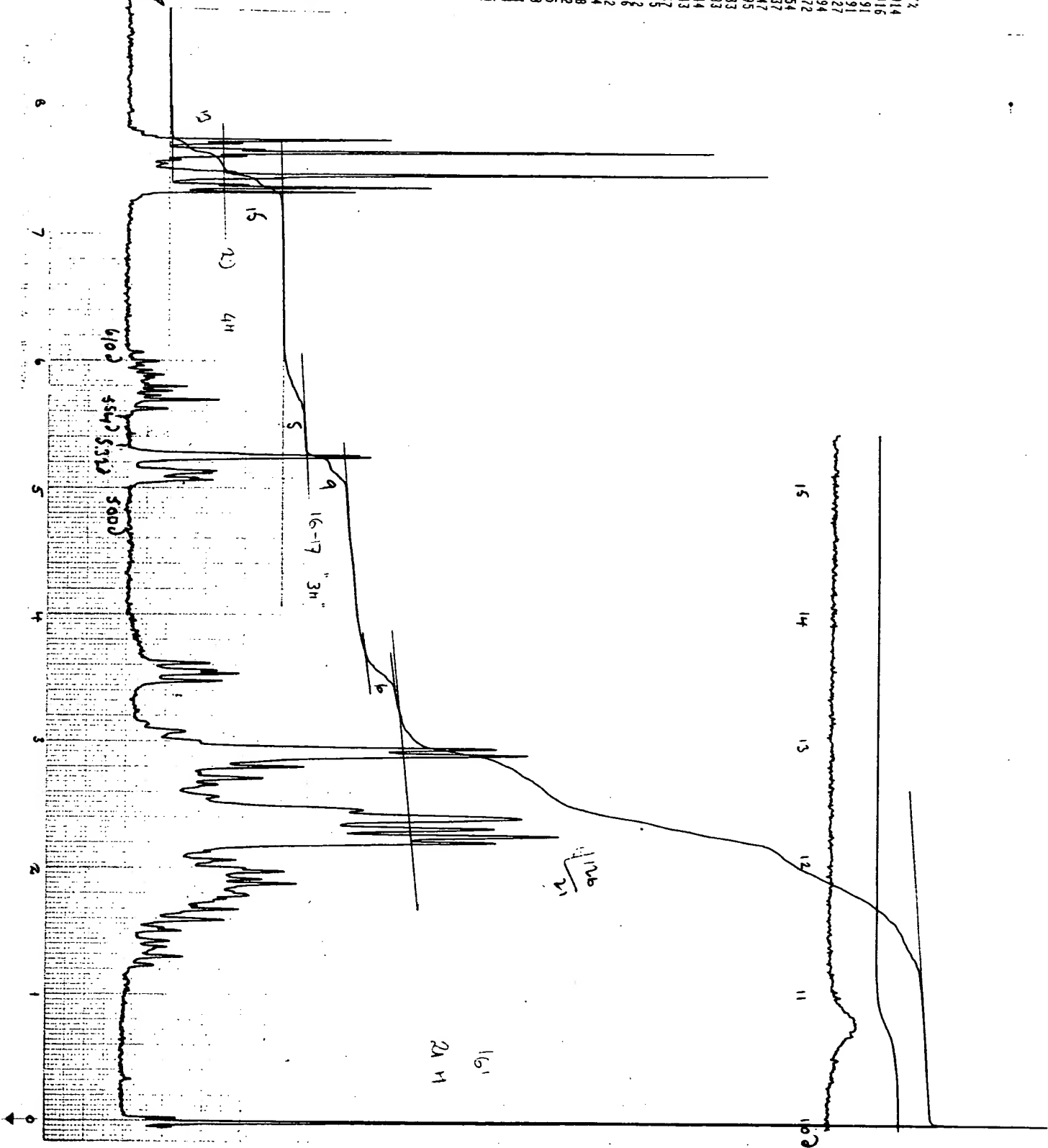
No peaks on offset

JEOL

Figure II

TAL 46
SOL 43751 -4 H2
REF 0.000PPM
31H 484.7500 HZ

FREQ(HZ)	PPM	INT
690.01	7.709	914
688.62	7.685	416
684.25	7.636	491
682.06	7.612	1991
679.87	7.587	427
668.58	7.460	394
666.31	7.436	2172
664.12	7.411	554
661.75	7.363	437
659.50	7.330	1047
657.25	7.304	795
518.00	5.781	233
514.93	5.746	183
511.87	5.712	144
508.81	5.678	343
503.88	5.619	187
498.96	5.529	852
493.93	5.421	336
487.59	5.363	272
482.37	5.323	318
323.75	3.613	324
318.50	3.554	342
316.31	3.530	415
311.06	3.471	360
258.33	2.883	1378
248.89	2.793	635
238.43	2.661	346
213.93	2.387	1357
206.93	2.309	1260
201.58	2.250	1483
196.87	2.197	1274
187.50	2.065	410
171.50	1.913	497
168.43	1.879	612
158.50	1.873	418
142.62	1.591	372
135.18	1.500	227
122.75	1.423	185
117.23	1.308	231
3.50	0.039	214
0.00	0.000	6532
-3.06	-0.034	200



Rc 82237

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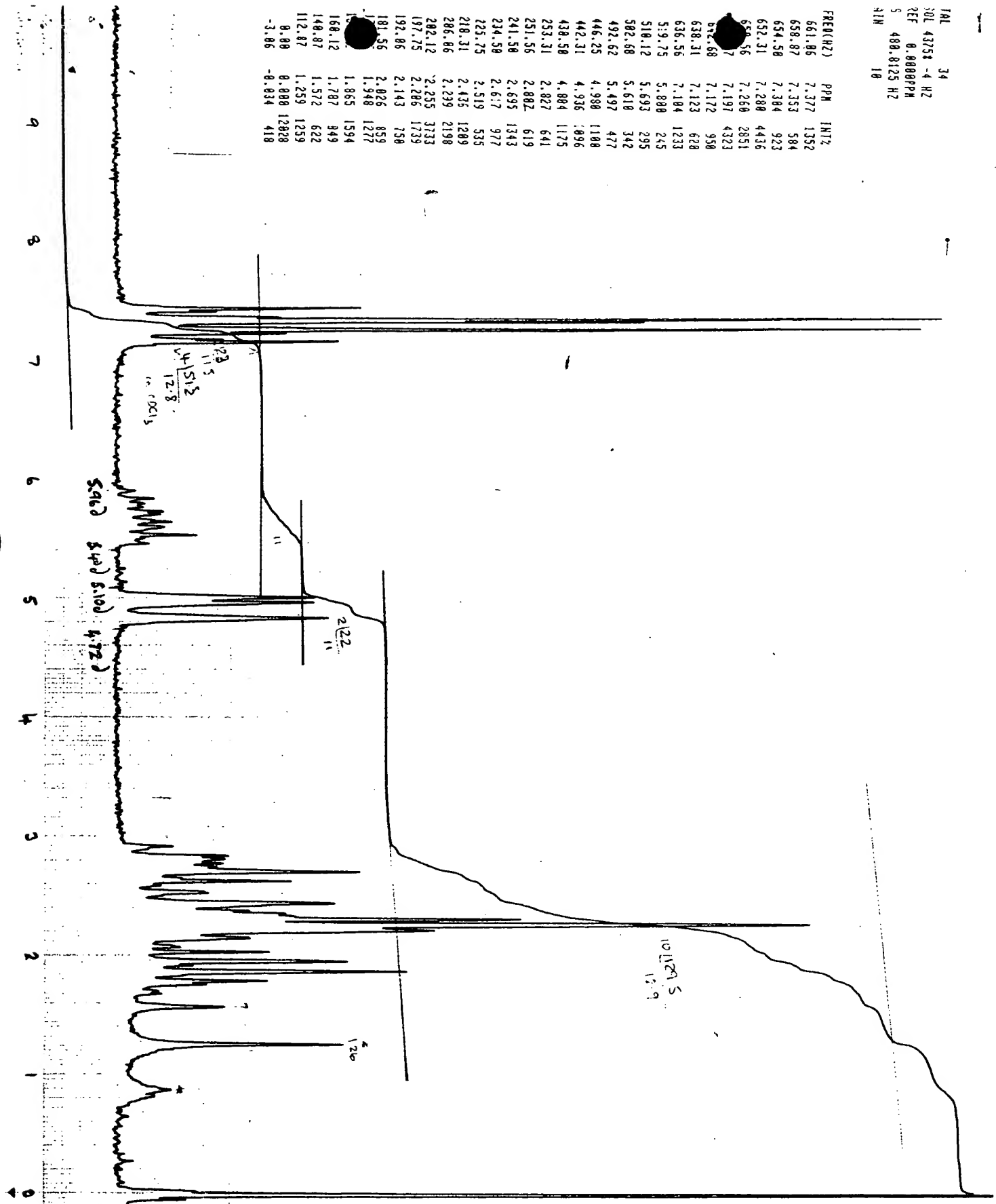
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Figure III

TAL 34
 SOL 43751 -4 H2
 REF 0.0000PPM
 S 480.8125 H2
 MIN 10

FREQ(HZ)	PPM	INT
661.86	7.377	1352
658.87	7.353	584
654.58	7.304	923
652.31	7.289	4436
650.56	7.260	2851
647.77	7.197	4323
646.68	7.172	958
638.31	7.123	628
636.56	7.104	1233
539.75	5.888	245
518.12	5.693	295
502.68	5.610	342
492.62	5.497	477
446.25	4.980	1108
442.31	4.936	1096
438.58	4.804	1175
253.31	2.827	641
251.56	2.802	619
241.58	2.693	1343
234.58	2.617	977
225.75	2.519	535
218.31	2.435	1209
206.86	2.299	2198
202.12	2.255	3733
197.75	2.206	1739
192.86	2.143	758
181.56	2.026	859
170.00	1.948	1277
168.12	1.865	1594
148.87	1.572	622
112.87	1.259	1259
8.08	0.000	12028
-3.86	-0.034	418



CDCl3
 TMS
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94.35
 20
 1792

11
 0.5
 3

418
 27.286

no peaks in offset
 4 g/cm3

JEOL